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***In Situ* Applications of X-ray Absorption Fine Structure**

Simon R. Bare
(Dow Chemical Company)

The ability of x-rays to penetrate matter was emphasized in this year's XAFS workshop on studying materials under *in situ* conditions. Eight speakers addressed the 60 attendees with topics ranging from catalysis to electrochemistry to biology. Jim McBreen (BNL) led off the workshop with a presentation on using XAFS to probe the electronic and geometric structure of fuel cell catalysts under applied voltages. He was able to draw a correlation between the Pt-Pt bond length and Pt d-band vacancies with reaction rates for several Pt-M alloy catalysts. Mark Antonio (ANL) described a novel bulk electrolysis cell for probing the structure of species in solution during electrolysis. The focus of the talk was on the $\text{Eu}^{2+}/\text{Eu}^{3+}$ couple. He showed, and explained why, the addition of a single f-electron to Eu dramatically changed the Eu L3-edge XANES. Simon Bare (Dow Chemical) followed the oxidation state of vanadium in a supported vanadium phosphate catalyst during the oxidation of butane using dispersive XANES with on-line product analysis. He showed a direct correlation between the presence of V^{5+} and the formation of maleic anhydride. The ability to study surfaces under corrosive conditions using XAFS was illustrated by Inho Song (Case Western Reserve). He described a total electron yield detector designed to perform *in situ* studies of the atmospheric degradation of materials. The state of sulfur in gaseous SO_2 in dry and humid environments was determined, together with that

on the surface of Cu films during exposure to the corrosive gas. Following lunch, during which several posters were presented, Di-Jia Liu (AlliedSignal) nicely demonstrated the need for true *in situ* experiments to study the form of Cu in a ZSM-5 de- NO_x catalyst. He showed that there is an excellent correlation between the presence of Cu(I) and the rate of selective catalytic reduction over this type of catalyst. A fascinating presentation on the use of micro-XANES to follow the uptake and metabolism of metals (Cr and Se) in aquatic plants was given by Doug Hunter (Savannah River Ecology Lab.). His data indicated that there is localized uptake and reduction of both elements. For example there is localized reduction of Se in the leaf of the fern from Se^{6+} to Se^{2-} . John Fulton (PNL) described both a high temperature (500 C), high pressure (1 kbar) reaction chamber for studying ion solvation in supercritical water, and a quartz capillary reactor for investigating metal species in supercritical CO_2 using EXAFS. He showed that there is a difference in solvation of Sr^{2+} and Rb^{+} ions in supercritical water compared to ambient water solutions: there is a reduction in the average coordination and a concomitant reduction in metal-oxygen bond length. The formal program ended with a presentation by graduate student Mike Nasher (U. of Illinois) on the use of *in situ* EXAFS to monitor the preparation of Re-Ir clusters on alumina prepared from novel bimetallic carbonyl complexes.



Opportunities in Polymer Research Using Synchrotron Radiation

Thomas P. Russell
(IBM Almaden Research Center*)

The use of synchrotron radiation for the investigation of polymers began in the early 1980's with exclusive use of small angle scattering methods to elucidate the morphological characteristics of bulk polymers. Despite the fact that the results from these studies were impressive, in quantity and quality, the growth in the application of synchrotron radiation for studying polymers was slow. Recently, however, there has been a tremendous growth in polymer related research. Not only has the number of experiments that are performed on polymers grown in that time, but the diversity of scientists performing research on polymers has grown. Initially, only those scientists whose major research interests were in polymers performed work in this field. Now, however, scientists with more traditional physics and chemistry backgrounds must be included. This growth can be attributed to several factors. First, there is the nature of the polymer chain. Typically, polymer chains pervade volumes on the tens of nanometers size scale. Thus, features characteristic of phase transitions are easily observable. Second, due to the entangled nature of the polymer chains, relaxation processes are longer. Thus, polymers are ideal candidates for the investigation of kinetic phenomena. Third, some of the potentials for using the variable wavelengths in the hard and soft x-ray region of the spectrum for the study of polymers have been realized. Thus, entire new avenues of research have been uncovered. Finally, the realization that the surfaces of polymers are quite smooth (an rms roughness of 0.6 nm) has permitted the exploitation of the numerous surface characterization techniques that have been developed at synchrotron sources. Both the increase in the number of scientists investigating polymers and the number of techniques available to pursue polymer re-

search have had significant impact on basic and applied research topics. A Workshop on Opportunities in Polymer Research Using Synchrotron Radiation was held during the 1996 NSLS Users' Meeting. It was designed to highlight some of the recent pioneering studies on polymers and to demonstrate areas of potential use of these versatile light sources in polymers.

Professor Benjamin Chu from the State University of New York in Stony Brook spoke on small angle x-ray scattering studies on polyelectrolyte gel/surfactant complexes. The low contrast between the scatterers, the low concentrations and the large size of the chains mandate the use of a very high resolution small angle spectrometer with high flux. These conditions can only be realized at a synchrotron source. The results from the experiments described by Chu have provided significant insight into the fundamental behavior of polyelectrolyte gels, the influence of the pH of the swelling solvent and the testing of different theoretical concepts.

One of the important developments that has occurred with synchrotron sources is the ability to perform several different measurements simultaneously, as for example small angle scattering and wide angle diffraction. Obtaining multiple results on a single sample has tremendous advantages in the case of polymers. Due to entanglements, it is often difficult to get two different polymer samples into precisely the same state and, consequently, errors are introduced when multiple samples are used. Simultaneously using multiple techniques eliminates this error. Dr. Benjamin Hsiao of DuPont discussed a series of studies where both small and wide angle x-ray diffraction were used to investigate the crystallization and melting of some semi-rigid polymers, namely poly(ether-ether-ketone), PEEK. Typical data obtained from such studies are shown in **Figures 1a and 1b** where the time resolved small angle x-ray scattering and the wide angle diffraction data for a PEEK sample are shown after the sample has been cooled from the melt to 290°C. In **Figure 1a** the small angle scattering data show a growth in a peak which is due to the crystallization of the PEEK molecules into a lamellar type of morphology. Simultaneously the diffraction data in **Figure 1b** show the development of the crystal structure in the lamellar morphology. Large anisotropic changes in the unit cell parameters occurred in these materials during secondary crystallization processes. By correlating the small and wide angle

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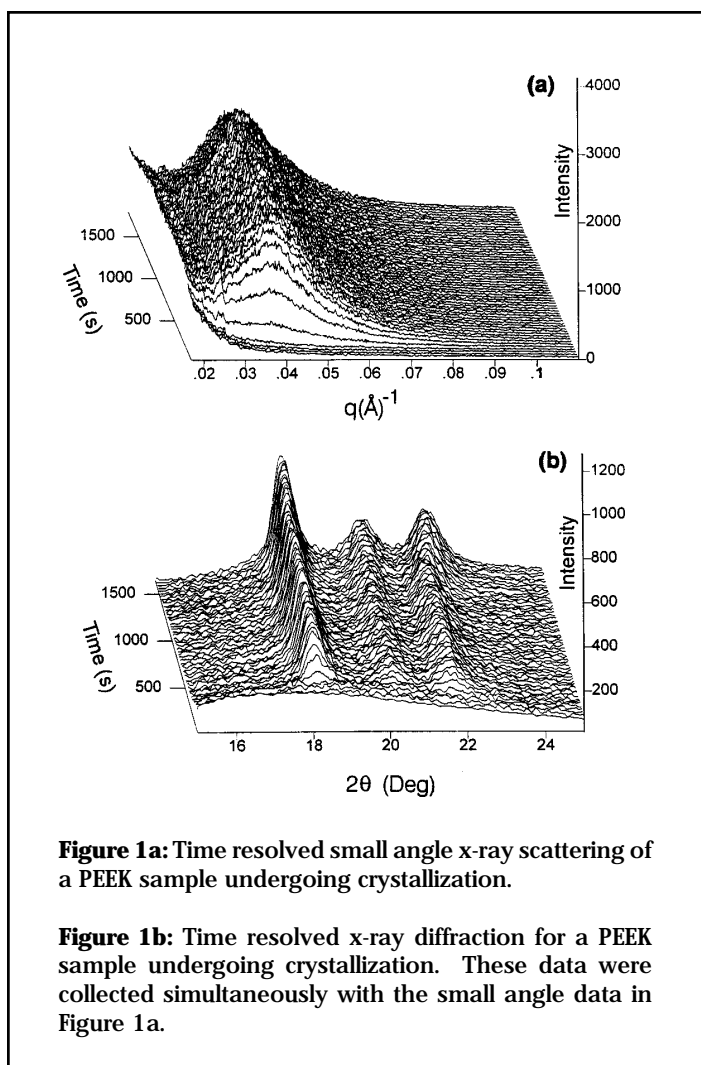
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scattering data a model for the crystallization process in PEEK was developed. Information of this nature is crucial in deriving structure property relationships which directly impact the processing and end use of polymers.

Another area where synchrotron radiation has played an important role is in the characterization of single fibers or small bundles of fibers. Using standard laboratory equipment the available flux is simply too small obtain acceptable signal to noise ratios. Professor David T. Grubb of Cornell University described experiments on high modulus polyethylene fibers, single fibers of Kevlar (about 12 microns in diameter) and bundles of spider dragline silk fibers ($\sim 4 \mu\text{m}$ in diameter) where the fibers are placed under a load and stretched. Within seconds, full

two dimensional fiber patterns could be obtained which permitted the investigation of dynamic processes and real-time crystal orientation. His lecture focused on deformation mechanisms that were dominated by elastic processes. Dr. N. Sanjeeva Murthy of AlliedSignal Inc. then described experiments on the fibrillar and lamellar structures in nylon-6 fibers. Again, with the ability to obtain full two dimensional patterns in seconds allowed the investigation of structural changes to the fibers during drawing and post thermal treatments. However, as pointed out by Murthy, since massive amounts of data are collected, much information has been ignored. He then described some of the efforts he has been making in the utilization of the full pattern.

The afternoon lectures in the workshop focused on the surface and interfacial behavior of polymers. Professor Miriam Rafailovich of the State University of New York discussed specular reflectivity and off-specular scattering studies on Langmuir films of ionic block copolymers at the air/water interface. Block copolymers, which are essentially two different polymers covalently bound together at one end, can be made such that one block is hydrophobic and the other is hydrophilic. On a water surface the difference in the functionalities of the two blocks cause the formation unique and unusual two dimensional structures. The lateral surface pressure dictates the concentration and, consequently, the morphology. These surface structures and the transition from one structure to another can be followed by use of off-specular scattering where a component of the diffraction vector is in the surface plane. However, the off-specular scattering is orders of magnitude less than the specular reflectivity and, consequently, requires the use of a synchrotron source to obtain good statistics. Rafailovich then demonstrated another area where off-specular scattering was being brought to use. This related to the conformality of the polymer films on a rough or periodic surface. **On the following page, Figure 2 shows** the rocking curve of a substrate that has a $1 \mu\text{m}$ grating on the surface. This spectacular profile was obtained by rocking the sample by an angle λ about the specular condition at $Q_z = 0.2 \text{ \AA}^{-1}$ (where the diffraction vector is oriented normal to



the surface plane of the sample). The multiple peak results from the periodic structure, i.e. the grating, on the surface of the sample. Coating this surface with a polymer film and then observing the time dependent changes in the off-specular scattering have yielded considerable insight into the motions of polymers on surfaces and the ability of polymers to planarize the free surface.

Following this, Dr. Thomas P. Russell of the IBM Almaden Research Center described a series of studies using Grazing Incidence X-ray Scattering (GIXS) to probe the structure of polymers near a surface. In the particular case of an aromatic polyimide, GIXS showed that, in the vicinity of a surface, order in the polymer was enhanced. Polishing or buffing of the polymer surface, a process used in the production of flat panel displays, was shown, by GIXS, to produce a strong orientation of the polymer chains in the vicinity of the surface in the buffing direc-

tion. This surface orientation is directly correlated with the orientation of liquid crystals in displays. Prefacing the next two lectures, Russell also described the use of Near Edge X-ray Absorption Fine Structure (NEXAFS), where the polarization of the incident x-ray beam was used to probe the orientation of the polymer chains at the surface. For example, shown in **Figure 3** are the NEXAFS results for an aromatic polyimide that has been buffed. By measuring the Auger and total electron yield as the energy of the polarized x-rays is changed where the electric field vector of the radiation is either parallel or perpendicular to the orientation direction, information concerning the surface (first 10 Å from the surface) and the near surface (about 0.70 Å from the surface) orientation of polymer chains is found. These results, combined with the GIXS results, have led to a detailed understanding of the buffing process and the manner in which information can be transferred across a boundary.

Much emphasis has been placed lately on the behavior of thin films of multicomponent polymer systems. Numerous techniques are available for depth profiling concentration in such systems, however, there is a paucity of techniques for investigating lateral heterogeneities. Professor Harald Ade of North Carolina State University described advances that he and his collaborators have made in the use of x-ray microscopy to this end. The advantage of x-ray microscopy over the more traditional techniques, like light microscopy and atomic force microscopy methods, is that different chemical components can be seen with a lateral resolution of 0.1 micron. In addition, since the x-rays are polarized, lateral variations in the orientation of

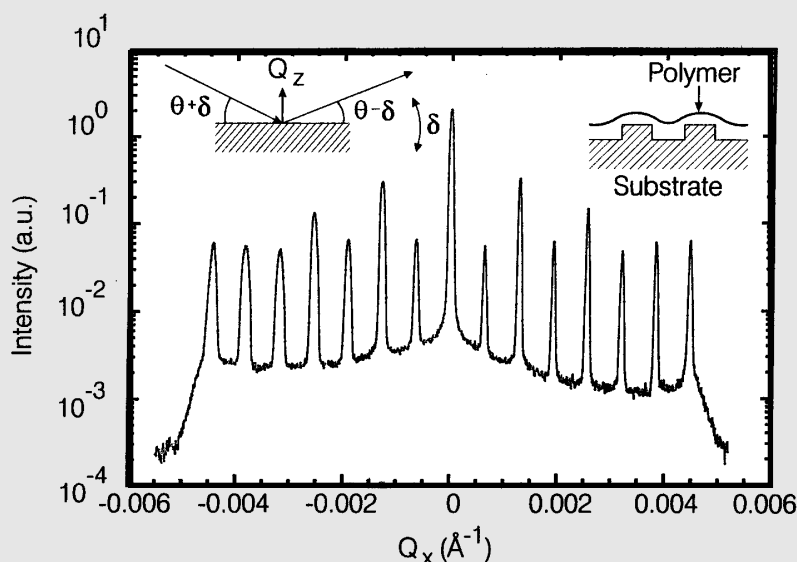


Figure 2: Rocking scan of a grating obtained at $Q_z = 0.2 \text{ Å}^{-1}$. With increasing angle of tilt of the sample with respect to the incident beam, there is a larger component to the scattering vector which permits the investigation of lateral heterogeneities on the surface. The peaks reflect the periodic nature of the grating.

the same or different components can be seen. Ade described investigations on liquid crystalline polymers, ternary polymer mixtures and polyethyleneterephthalate where x-ray microscopy has provided key insight into the morphology and orientation. X-ray microscopy is relatively new but, as explained, the number of microscopes available for study is growing at a rapid rate. In the study of the surface and interfacial behavior of polymers it is increasingly more important to distinguish the behavior of the polymers immediately at the surface or interface

without having to extract such information from a technique that averages over the first 5-7 nm. In the case of polymers, at a distance of only several monomer diameters from the surface, about 1-2 nm, the bulk characteristics of the polymer chain are found. Dr. Benjamin Dekoven of the Dow research laboratories described studies using near edge x-ray absorption spectroscopy on materials ranging from self-assembled monolayers to perfluorinated polymers where chemical information was obtained on the first nanometer from the surface. Such information is

crucial for developing a quantitative understanding of the surface wetting characteristics of polymers and the transfer of information, be this chemical or structural, across an interface.

Each of the lectures focused on specific research problems. However, there were several underlying messages that became quite clear as the workshop progressed. First, synchrotron radiation sources offer a tremendous battery of techniques for the characterization of polymers. The results presented beautifully demonstrated several examples where quantitative information was obtained. Second, the number of experiments performed at synchrotron sources with polymers as the central research theme has increased tremendously over the past few years. Finally, there is still much that these sources offer for performing polymer related research, a resource that will lead to pioneering developments in the field.

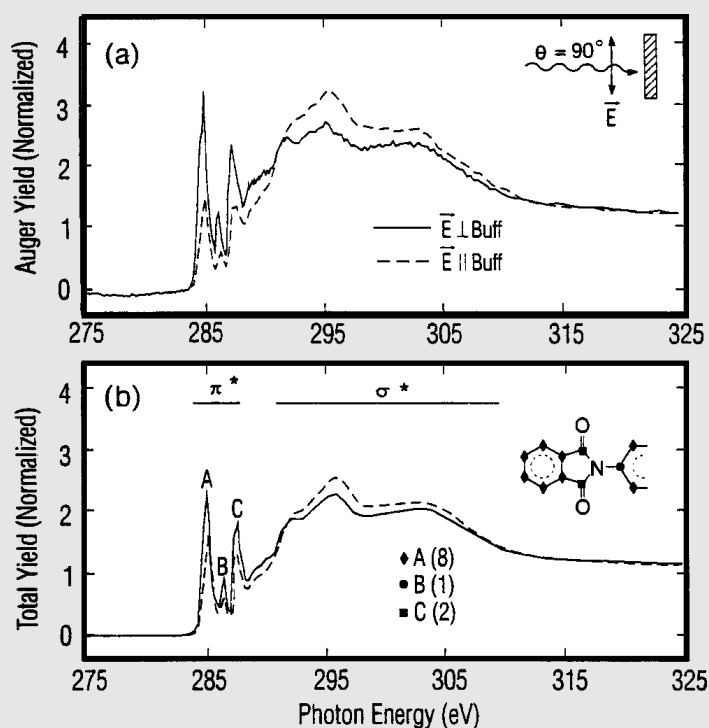


Figure 3: Near Edge x-ray absorption fine structure for an aromatic polyimide near the carbon edge where the polyimide has been oriented at the surface. Both the Auger and total electron yield are shown with the electric field of the x-rays parallel and perpendicular to the orientation direction of the sample.

Multiple Wavelength Anomalous Dispersion (MAD) in Protein Crystallography

Lisa J. Keefe and Stephan L. Ginell
(Argonne National Laboratory)

Multiple wavelength anomalous dispersion (MAD) has developed into a powerful crystallographic phasing technique for solving macromolecular structures. Synchrotron radiation is essential in order to implement MAD phasing, and with the increasing accessibility of synchrotron beamlines, the technique is gaining widespread use. The focus of this workshop was to present and discuss the theoretical approaches to MAD phasing, the preparation of selenomethionyl-substituted protein for MAD phasing, the strategies of MAD data collection, processing and scaling, and examples of the application of MAD phasing in solving crystal structures.

Axel Brunger (Yale University) presented a comparison of algebraic and probabilistic methods applied to a MAD data set of a fragment of the carbohydrate binding domain of mannose binding protein A. Tom Terwilliger (Los Alamos National Laboratory) discussed a Bayesian approach to MAD phasing, as implemented in his program HEAVY (v 4.5). In a comparison of Bayesian MAD phasing with other methods, namely treatment of MAD data as approximate relationships of dispersive and anomalous differences among wavelengths and treatment of MAD data as multiple isomorphous replacement, Bayesian MAD phasing gives more accurate phasing, especially for incomplete data sets. Alan Friedman (Purdue University) discussed data reduction and scaling for MAD, the inherent problems with MAD data and solutions to those problems, and MAD phase determination. With MAD data, the signal is inherently less than the noise and data collation can be problematic. To overcome these two obstacles, local scaling with flexible automatic data collation can be performed and robust phase determination can be used.

Robert Sweet (BNL-Biology) presented a talk for Venki Ramakrishnan (University of Utah School of Medicine) on the use of standard MIR data reduction, scaling, and phasing programs to analyze MAD data. Dan Leahy (Johns Hopkins School of Medicine) discussed his experiences with the use of selenomethionyl-substituted protein to solve structures by MAD. He reviewed strategies for substituting selenomethionine for methionine in proteins, methods for growing bacteria in media containing selenomethionine and expression of selenomethionine-substituted protein, tips on purification and crystallization of selenomethionine-substituted protein, assays for selenomethionine in purified proteins, and valuable advice on how to prepare for and perform a MAD experiment at a synchrotron. Robert Sweet (BNL-Biology) discussed issues in data collection for MAD phasing, with much emphasis on the integrated and user-friendly beamline-control software used at his beamline at NSLS.

Chi-Hon Lee (Rockefeller U.) presented the crystal structure of HIV-1 Nef complexed with a src-family SH3 domain. Yee Liu (Columbia U.) discussed a MAD experiment at the M4 edge of uranium. This is a very difficult and unique experiment due to the low energy (3728 eV) of the M4 edge of uranium. The advantage of using this heavy atom is that the MAD signal is very strong and thus phasing can be accomplished for very large molecules or complexes.

The workshop ended with a lively question and discussion session which focussed on the details of solving structures using MAD phasing. These included the likelihood of solving a structure using MAD phasing. This is correlated with the signal to noise ratio, and methods for calculating this ratio were reviewed. Also discussed was the determination of the anomalous and dispersive components, f'' and f' , respectively, of the structure factor and the refinement of these components, with and without anisotropy. MAD phasing is rapidly becoming the technique of choice for the determination of crystal structures and the increasing availability of synchrotron radiation beamlines and development of cryocrystallographic techniques for performing MAD experiments is enabling many otherwise very difficult crystal structures to be solved.



Strategy and Methods for Area Detector Data Collection

Alex Darovsky
(Brookhaven National Lab., NSLS)

The rapid progress in the technology of area detectors (AD) increases their advantages for many scientific applications utilizing x-ray diffraction techniques. Being primarily oriented to macromolecular studies, AD data collection techniques and subsequent reduction and interpretation of the data need to be readjusted for the new applications, particularly for small molecule crystallography, powder diffraction, and synchrotron crystallography.

This one day workshop was intended as a forum for the discussion and evaluation of methods, procedures, and algorithms for AD data collection and processing. The morning session was dedicated to in-depth analysis of methods for 2D data processing; the afternoon session being focused on practical aspects of 2D data collection strategy. Following opening remarks by NSLS Chairman Michael Hart, Robert Bolotovskiy (SUNY @ Buffalo) gave a detailed description of a new method for intensity integration of 2D diffraction peaks. He emphasized the advantage of using the "seed-skewness" method for integration of weak peaks or some abstinent peaks with irregular shapes.

Joseph Reibenspies (Texas A&M U.) discussed the topological property of 2D arrays of data with implication to peak/anomaly classification of diffraction pattern - another example of generalization of data reduction procedures for the AD technique. Finally, analysis of a severely delocalized diffraction pattern in terms of its crystalline and amorphous components was demonstrated by Peter Moran (SIEMENS). He applied a Fourier filtering technique for estimation of crystallinity of partially oriented polycrystalline samples.

The afternoon session, chaired by Richard Harlow, began with a detailed analysis of geometrical conditions inherent to the oscillation method presented by Robert Sweet (BNL-Biology). The impact of the presentation was greatly enhanced by remarkable drawings by co-author, Zbyszek Dauter (EMBL, Germany). An advocate of image plate technology, Wladek Minor (U. of Virginia) gave an extensive overview of recent developments in area detector systems and auxiliary x-ray equipment which enhanced the capability of in-lab diffractometers making them competitive with the existing synchrotron sources. His talk also focused on the practical aspects of data reduction, particularly, on the analysis of possible pitfalls and adequate criteria for data quality. The first two speakers referring predominantly to macromolecular crystallography were complemented by aggressive enthusiasts from the small moiety community. Patrik Carroll (U. of Pennsylvania) demonstrated a superb performance of the R-AXIS image plate system for accurate structure analysis. A vast array of interesting molecular structures illustrating his presentation exemplified the effective and professional approach in utilizing the commercial hard- and software systems. A competitive, also an expensive so far, utilization of a CCD detector for single crystal data collection and processing was introduced by James Phillips (SIEMENS). While offering a versatile set of sophisticated hardware, he stressed the importance of optimization of experimental technique in order to obtain the best quality data. A detailed description of optimized strategy and tools implemented in SMART SYSTEM was given and evaluated.

Finally, Fabienne Adolphe (ESRF, France) reviewed the experience accumulated at the BL2 beamline (ESRF) with area detectors. The variety of approaches which were reported indicates that synchrotron sources, being multi-user facilities with diversified areas of research, provide a fertile field for further applications and development of area detector techniques as was manifested by the superb attendance at the workshop. Comprehensive Workshop Notes (partially supported by DuPont de Nemours Co. and SIEMENS) made a valuable addition to the educational objective of the workshop.



The Application of Synchrotron Radiation to the Study of Magnetic Materials

Boris Sinkovic
(New York University)

The workshop brought together a diverse group of researchers involved in the application of a variety of synchrotron radiation (UV and x-ray) based techniques (scattering, spectroscopy and microscopy) to study of magnetic phenomena in magnetic thin films and multilayers, interfaces, surfaces, and low dimensional magnets. Gary Prinz's (NRL) talk during the NSLS Users' Meeting, a day before the workshop, describing applications of variety of novel magnetic structures for storage, sensors and logic technologies, was an excellent introduction to the workshop. The workshop's opening remarks by Denis McWhan's (BNL) reiterating the increased interest and activity in studies of magnetism in recent years, which were also reflected in larger than anticipated workshop participation, resulted in a need to move the workshop from Berkner Hall to the NSLS Seminar Room.

The first talk was given by Bernhard Keimer (Princeton U.) who discussed x-ray diffraction experiments on compounds undergoing structural transitions induced by high magnetic fields. These studies led to the discovery of a novel "soliton lattice" phase in the quantum spin-chain compound CuGeO_3 , which provide an important test for theories of low-dimensional quantum magnetism. Gavin Watson (BNL - Physics) reported on recent progress in applying resonant x-ray scattering techniques to study the temperature-dependent magnetic order near the surface of an antiferromagnet. The studies show that within $\sim 50 \text{ \AA}$ of the (001) surface of UO_2 , the magnetic scattering intensity decreases continuously as the bulk N'el temperature is approached from below, which is in contrast to the bulk transition. Rogerio Paniago (U. of Houston) talked about x-ray diffuse scattering study of interfacial roughness in metallic multilayers using a novel 2-dimensional Image Plate detector. The technique allows the measurement of the conformal rough-

ness in the initial stages of growth of a lattice matched Fe/Au buffer and the height-height correlation function. The interfacial cutoff length was shown to be dependent on the lattice mismatch, indicating it to be an important parameter for the growth of high quality metallic multilayers. Stuart Parkin (IBM Almaden) talk gave several examples how his work in thin film magnetism has benefited by the application of synchrotron radiation techniques, in particular EXAFS and MCD. Yves Idzerda (NRL) talked about recent development in the absorption and reflection of circularly polarized soft x-rays demonstrating the ability to obtain element-specific magnetic information for heteromagnetic thin films, including a means to determine the magnetic structure, to measure the film thickness with very high accuracy, to identify sequence of multilayer switching with applied field, and possibly to determine the magnetic roughness of an interface.

In the afternoon session Elio Vescovo (BNL- NSLS) showed, through various examples, how a combination of spin- and angle-resolved valence-band photoemission spectra and *ab initio* slab calculations can yield a very detailed description of the electronic structure of magnetic surfaces, interfaces and thin films. Such studies reveal the precise relation between the electronic structure and the magnetic properties. Jaehoon Park (BNL-NSLS) talked about electronic aspects of the ferromagnetic transition in materials exhibiting colossal magnetoresistance (CMR). The high-resolution photoemission data found the insulator-metal transition near the Curie temperature and suggesting that strong small-polaron effects are responsible for the enigmatic insulating behavior above T_C . Neville Smith (LBNL) discussed opportunities for magnetic materials research at that high brightness synchrotrons, like ALS, with special emphasize on the microscopic studies. Ulrich Hillebrecht (U. of Duesseldorf) talked about element specific magnetic imaging with Photoelectron Microscopy using a commercial, Staib PEEM-150 microscope. Finally, Andrew Kent (NYU) summarized his research on the fabrication and study of nanometer scale ferromagnetic structures prepared using a scanning tunneling microscope. The work demonstrates that geometrical similar iron nanostructures are magnetically distinct, indicative of important contributions to the magnetic anisotropy other than the shape.



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Hard X-ray Precision Fabrication

D. Peter Siddons and Erik Johnson
(Brookhaven National Lab., NSLS)

This workshop was intended to be a true workshop rather than a mini-conference. It was our intention to bring together people who are active in fabricating lithography-based structures in order to stimulate discussion around the topic area, and to seek advice on how to proceed in our development of the X14B beamline as a manufacturing research end-station for hard x-ray fabrication. There were approximately 15 attendees. Judging from the level of enthusiasm among the attendees it would seem we met with reasonable success in these objectives. There were five key areas of concern regarding this program which were addressed at the meeting:

- Support Capabilities
- Exposure Station Properties
- Interoperability with Other Facilities
- Qualification of Customer Vendors and Tools
- Beamline Access and Allocation

The resulting discussion of each of these areas is described briefly below.

Support Capabilities:

It was felt by many at the workshop that some supporting laboratory space be available which would provide a clean area to assemble fixtures and package samples for shipment. At present we believe this can be met by cooperative use of the existing cleanroom in the basement of instrumentation, and by allocation of some of the X13 Lab space for setup. A measurement metrology capability is also regarded as desirable, and in this respect, our measurement microscope system (Nikon microscope plus the Quadracheck QC-4000 measurement package) was deemed to be adequate to the present state of process development.

A sample development capability was also requested. Currently two developer systems are in use, GG and

MIBK. Although some attendees favored the GG system, our research here indicates that for high aspect ratio material processing the MIBK/EtOH system seems to perform as well or better than the GG system. We would only consider the GG system if one of the users was sufficiently motivated to organize and fund its implementation.

Exposure Station Properties:

In our presentation we described the properties of our planned scanner. There was general agreement that a high linear velocity device was desirable. Our prototype hydraulic stage will be capable of scan rates up to 25cm/s with position and speed servo-feedback. One key feature which was strongly advocated was interoperability of fixturing with other facilities. Since at present, no other facility is performing hard x-ray exposures it is largely up to us to establish a standard configuration in consultation with potential future facilities. Most notable among these in the USA is CAMD, who are adding a wiggler to their machine for this type of work. The scanner enclosure should have a controlled environment which includes class 100 particle counts, less than 50% relative humidity, electrostatic reduction, and temperature stability the order of 1°C . Ideally the scanner should be capable of exposing a 15cm by 15cm mask without step and repeat motions. The space constraints at X14B are such that we can only expose a field about 12 cm wide. This was viewed as a satisfactory step in the right direction from our present 4cm field at X27B.

Interoperability with Other Facilities:

This issue extends beyond fixturing and facilities. To make this a viable production technology, it was strongly felt that in the long run a μ print shop-capability should be created. In essence the idea is that customers would take their work to a broker (like MCNC) who

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would then distribute the masks, fixtures, and materials to whatever exposure station was suitable and available. This implies some dovetailing of scheduling of maintenance and possibly redundancy within facilities. In this way if a scanner goes down another is available as backup. At the NSLS, beamline X27B might, for example, serve as backup for X14B. In the case of facility maintenance, ALS, CAMD and NSLS might coordinate shutdown activities.

Although it seems early these days to be too concerned about this issue, there was a general awareness of its longer term impact on the viability of hard x-ray machining as a manufacturing technology. It was pointed out that this problem is not as acute as it is for the specialized analytical work of the type ALS and SSRL are undertaking for wafer analysis. In their case full time availability is essential to recover from manufacturing line problems. In our case, a brief unavailability is part of the normally expected contingency in any sound production plan. Still it is an issue worth noting.

Qualification of Customer Vendors and Tools:

If the technology proves both successful and popular, a concern was raised about liability, or at least culpability, for production runs that don't work out. If a customer provides a mask or fixture that won't work, or sub-specification material that cannot be properly processed, establishing responsibility for a failed production run could prove both difficult and contentious. This should not be a problem with our first users, who will essentially be collaborators. If the print shop model takes hold we will need to establish standards for acceptable material and qualified vendors of masks, fixtures, etc.

Beamline Access and Allocation:

Several questions arose which, again, are somewhat in the future regarding access to the station currently under construction at NSLS. The attendees of the meeting predicted that within a year of establishing the production research line, it will be oversubscribed by as much as a factor of 10! While NSLS would be pleased with such a success, the administrative issues must be established well in advance of such a demand. NSLS is seeking advice on these issues from the DOE.



